PREPARATION OF BIODIESEL AND SYNTHESIS OF NANOPARTICLES FROM *Calotropis procera* PLANT SEEDS AND LATEX

SYNOPSIS

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1. Introduction

As of late, the universal energy and request framework are confronting number of difficulties. With the world all out utilization of oil arriving at 84 million barrels for every day in 2005, the realized oil holds are assessed to be exhausted in under 50 years, at present pace of utilization, by 2.7% every year. Fumes outflow from transport engine vehicles utilizing petroleum products is one of the most significant issues adding to contamination and a worldwide temperature alteration. The lessening flexibly of petroleum derivatives, climate mindfulness, and expanding energy utilization and costs are the essential components prompting the quest for elective wellsprings of energy to supplant oil-based fills. In this manner, innovative work on elective powers is seriously revealed by scientists everywhere on the world to meet the future needs of energy universally (Taufiqurrahmi & Bhatia, 2011).

These days, bio-diesel is considered as one of the substitutes for oil inferred diesel (petrol/diesel), since it has frequent focal points in its overlap, for example, sustainable, biodegradable, nontoxic and diminished emission. Bio-diesel (unsaturated fat methyl esters, FAMEs) is gotten from transesterification of different vegetable oils. Notwithstanding, the bio-diesel creation is costly because of the excessive cost of crude materials, impetus choice, energy serious decontamination and partition of items. The ongoing examination has zeroed in the utilization of non-eatable vegetable oils, for example, *Calotropis procera* (Rengasamy et al., 2016)

A plant of the semi-dry jungles and subtropics, it is discovered developing at heights from ocean level to 1,300 meters. It fills best in zones where the mean yearly precipitation is in the reach 300 - 400mm. Requires a radiant position. There are leanings toward disorderly sandy soils. Plants that have been set up are drought resistant and salt tolerant to a high degree. It prefers and is frequently found in areas where construction has been abandoned, especially sandy soils in low-rainfall areas. When filling in sandy soils, this species has been known to escape from cultivation. The plant has spread generally from its unique reach and has gotten naturalized in numerous territories of the jungles and subtropics. Through its breeze and creature scattered seeds, it rapidly gets set up as a weed along degraded side of the road, tidal pond edges and in overgrazed local pastures. The plant is frequently a marker of overgrazed land (Sola & Olubummo, 2019)

Present day bio-energy advances and bio-fuels are generally considerate from ecological view point and produce almost no contamination if consumed accurately and totally. The

formation of new work openings inside the network and especially in rustic zones is one of the significant social advantages from the misuse of biomass for energy, industry and climate. The use of biomass for energy and industry allows a significant number of hydrocarbons to be consumed without raising the CO_2 content of the environment, thus showing a strong contribution to the Greenhouse effect and problems of "worldwide transition" as they arise in both developed and non-industrial countries. Other benefits of using biomass include the fact that biomass-derived fluid powers contain no sulphur, avoiding SO_2 outflows and lowering NO_2 emissions. Improved agronomic practices across all managed biomass farms would also lead to ecological improvement by assisting with soil balancing and preventing desertification, which is currently occurring (Henning & Gower, 2012).

The different kind of methods such as pyrolysis, catalytic cracking, blending and micro emulsification have been used to produce biodiesel from vegetable oil for diesel engines. Finally, transesterification process was developed as the most suitable method to overcome problems due to direct use of oil in diesel engines (Abbaszaadeh et al., 2012). Transesterification measure, as appeared in Figure 1 is an ordinary and the most well-known strategy for bio-diesel creation. In transesterification response homogeneous stimuli (antacid or corrosive) or heterogeneous stimuli can be utilized. The stimuli split the oil into glycerin and biodiesel and they could make synthesis simpler and quicker.

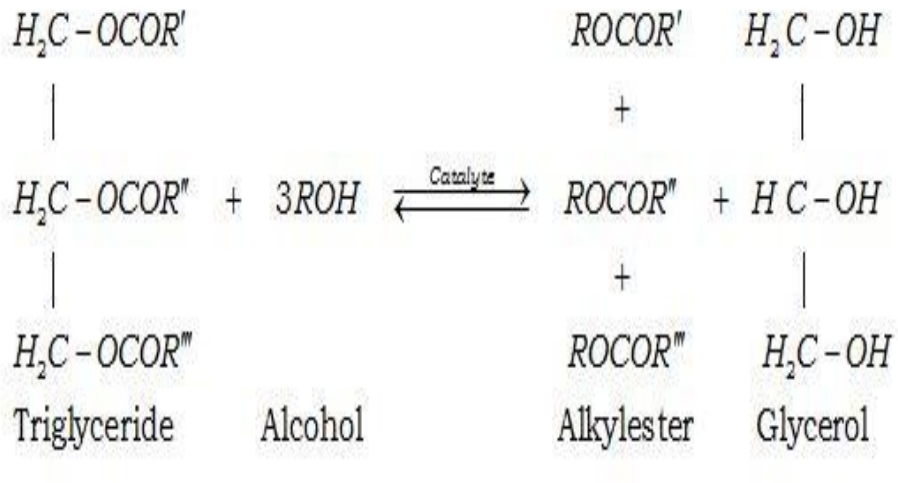

Figure 1. Bio-diesel preparation via transesterification reaction

In this strategy, unsaturated fat alkyl esters are created by the response of fatty substances with a liquor, particularly ethanol or methanol, within the sight of antacid, corrosive or compound stimulus and so on. Sodium hydroxide or potassium hydroxide, which is broken

up in liquor, is commonly utilized as stimulus in transesterification response. The results of the response are unsaturated fat methyl esters (FAMEs), which is the bio-diesel, and glycerin. Ethanol can be likewise utilized as liquor rather than methanol. In the event that ethanol is utilized, unsaturated fat ethyl ester (FAEE) is delivered as item (Henning & Gower, 2012). Consequently, creation of bio-diesel is getting more normal on a business scale utilizing sodium methoxide as catalyst because this catalyst helps maintain low moisture content. The clarification is that while utilizing an alkoxide, for example, sodium meth-oxide as stimulus, the water-forming reaction occurring at the same time is avoided.

$ROH + XOH \Longrightarrow ROX + H_2O$, (R = alkyl, X= Na, K)

Toward the start of the transesterification response, the combination of vegetable oil and methanol comprises of two immiscible stages, the vegetable oil and the liquor. This prompts a circumstance where mass exchange might be restricting the general response rate instead of the real substance response. Also, toward the finish of the response two products are formed, the methyl esters and glycerol as coproduct. The two phases can be easily separated by gravity (settling of the stages) or centrifugation however the last is energy concentrated. To eliminate excess impetus and possible foreign substances, such as glycerol produced during the reaction, the product ester phase must be washed with water. The transesterification happens stepwise, continuing from the triacylglycerol beginning material by means of the di-and monoacylglycerols to glycerol, with methyl ester being framed at each progression. Although under ideal conditions, the transesterification response continues to an extremely serious level of change, minor to follow measures of triacylglycerol beginning material just as mono-and diacylglycerols remain. Indeed, even minor measures of a portion of these materials can significantly influence fuel properties (Knothe & Razon, 2017).

Calotropis procera has one more application, we can synthesize the nanoparticles from the milky-latex of *Calotropis procera*. The milky latex of *Calotropis procera* has great potential for synthesis of nanoparticles like iron, copper, and nickel etc. These nanoparticles play a very important role in the production of biodiesel also they act as a catalyst in the transesterification reaction (Harne et al., 2012). Nanoparticles are tiny particles with a size scope of 1–100 nm. Broad examination has been led for the combination and use of nanoparticles over the previous decade. Nanoparticles assume a huge function in various fields, for example, food, feed, drugs, and beautifiers dependent on their uncommon qualities. Nanoparticles have just been demonstrated to have a significant part in human wellbeing improvement, food, and horticulture. What's more,

they are likewise utilized in the treatment of non-medicinal innovation. Organic union is a naturally agreeable, cost-effective, nontoxic methodology for the creation of nanoparticle. Copper nanoparticles are gaining critical ground in the zone of nanotechnology and nano medicine for a very long time in view of their magnificent synergist, optical, electrical and anti-fungal antibacterial application (Harne et al., 2012). NiO NPs has wide scope of uses like battery cathodes, photograph electron gadgets, particle stockpiling material, sensor, for example, gas sensor, attractive materials, thermoelectric materials, impetuses, energy components, necklace sharpened photo-cathodes, electrochromic films, anticancer, cytotoxic movement and non-enzymatic glucose sensor and so on, NiO NPs additionally displays antibacterial action against a gram-positive and gram-negative microbe. Among the different substance and actual strategy for getting ready nanomaterial is green amalgamation technique that draws the attention of scientists. Since, this technique for green blend has the points of interest like basic, cost effective, requires less temperature, energy, pressure and earth cordial (Anand et al., 2020).

Iron oxide nanoparticles are one of the most eco-accommodating particles and these are combined in mag-hematite, magnetite and hematite structures, through natural, physical and synthetic strategies. Bio-active components, present in green plants, result in many applications in combination with nanoparticles. In this investigation, endeavours were made to advance the amalgamation of magnetite iron oxide (Fe_3O_4) nanoparticles, utilizing *C. procera* leaf extract, as a reducing agent. Nanoparticles will be characterized using Fourier transform infrared (FTIR) spectroscopy, X-Ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersion X-Ray (EDX). *Calotropis procera has* the great importance for the production of bio-diesel from its seeds and synthesizes of nanoparticles from its milky latex. The figure of *Calotropis procera plants* is given below as Figure 2.

Figure 2: - (A) *Calotropis procera* plant (B) Seeds of *Calotropis procera* Plant samples will be collected at Kathua district, Jammu & Kashmir, India, 184204.

2. Literature Review

Exploration on elective powers remained generally torpid in the years after World War-II until the energy emergencies of the 1970s spurred recharged interest in this subject. In this association, vegetable oil was rediscovered as expected energizes for diesel motors. The likely first covers esters of vegetable oils from this time date to 1980. In this work, scientists from South Africa depicted the use of methyl esters of sunflower oil in a diesel motor and that the utilization of these esters reduced the thickness issue and resulting operational issues (Knothe & Razon, 2017). In 2011 Nikken Taufiqurrahmi and Subhash Bhatia had found that the synergist breaking may be the most efficient methodologies to break acceptable feed-stock inside seeing catalyst to take bio-fuel (Huber & Croma, 2007). Close by tasteful vegetable oils, non-consumable and used cooking oils have got huge thought as feed-stocks for the making of bio-fuel. The synergistic breaking of attractive and non-consumable oils requires the headway of fitting breaking stimuli and reactors for the formation of bio-fuel, for instance, bio gasoline. Ali Hassan Ibrahim developed a situation, in light of artificial natural product dehiscence, to gather seeds and silk fibers of *C. procera* with no disaster (Huber & Croma, 2007). The seeds and floss got by this strategy were contrasted and those gathered normally. Regular organic product dehiscence caused a stamped decline in seeds and fibers yielded from *C. procera*. The seeds and fibers acquired under artificial organic product dehiscence were roughly six fold higher than those got under regular natural product dehiscence (Ibrahim et al., 2016).

In 2016 the Mookan Rengasamy had observed that the castor leaf extricates mediated measure for the blend of iron nanoparticles and their reactant ability in the transesterification of castor oil for the creation of bio-diesel. The photosynthesized iron nano catalyst was described by spectroscopic and electron tiny examination. The significant physical and compound properties of the bio-diesel were additionally separated according to American society for testing and materials (ASTM) (Rengasamy et al., 2016).

In 2018 the researcher Lundeberg Ventura DeSousa gave a focus on *Calotropis procera plant* and found that the *C. procera* oil presents a decent potential to be utilized in the union of bio-diesel, showing that it is important to develop the exploration on the employments of *C. procera* for making biodiesel (Sousa et al., 2018). So, the bio-diesel is a very important substituent of diesel. Declining oil assets, joined with expanded interest for oil by arising economies, just as political and ecological worries about petroleum derivatives

are driving our general public to look for new wellsprings of fluid energizes. The lonely current sustainable source of natural carbon is plant biomass, and bio-fuels derived from plant biomass are the only current sustainable source of liquid fuels. Biomass is an economical, inexhaustible, and plentiful source of carbon (Huber & Croma, 2007).

Other than planning biodiesel, several different methodologies have been examined over the long run with the goal of decreasing the thickness of triacylglycerol-containing materials. These methodologies decrease quality of Petro diesel, microemulsification, and pyrolysis (Anand et al., 2020). Besides these four methodologies, all the more as of late another methodology dependent on a synergistic response named hydrodeoxygenation, which bears the cost of a combination of hydrocarbons most likely best called "sustainable diesel" furthermore, re-enacting the organization of oil determined diesel fuel (Petro diesel), has been created (Knothe & Razon, 2017), (Huber & Croma, 2007). The current article is, notwithstanding, worried about bio-diesel and different methodologies are not examined here. Along with it we had also reviewed the various studies on the nanoparticles synthesized by *Calotropis procera* milky latex. The latex of *Calotropis procera* will be used for the production of nanoparticles like iron, nickel, and copper through green methods. In spite of the fact that nanoparticles are associated with contemporary science, they have a long history. Nanoparticles were utilized by craftsperson as far back as Rome in the fourth century in the celebrated Lycurgus cup made of diachronic glass, just as the ninth century in Mesopotamia for creating a sparkling impact on the outside of pots. Bunches of scientists have done research in the arrangement of metal NPs alongside their application (Scheming & Remita, 2015). Synthetic strategies chiefly include decrease through synthetic compounds by holding salvo warm or aqueous procedure, sol-gel technique, polyol strategy, and so on (Zhang et al., 2002), (Charitidis et al., 2014). Be that as it may, compound responses are hazardous, expensive, abrogating high energy and hard proportional up. Right now, there is a developing need to create Eco-accommodating movements, which don't utilize harmful synthetics in the blend shows. Green combination as a natural methodology incorporates blended valence polyoxometalates, polysaccharides, Tollens, organic, and illumination procedure which have pay over regular techniques including substance arbiters related with ecological harmfulness. Determination of dissoluble medium and choice of Eco-accommodating nontoxic lessening and settling specialists are the main issues which essentially be considered in green blend of NPs. Here is an extraordinary need to make eco-friendly and financially practicable revolutions for material incorporation encouraged the quest for bio mimetic strategies for union

(Kalishwaralal et al., 2008). So, organic strategy is straightforward simple and cut-rate technique for the development of NPs (Iravani, 2011).

From the above literature we observed that the bio-diesel from *Calotropis procera* plant seed oil is reported in very few studies. The latex of this plant will be used for the production of nanoparticles through eco-friendly green methods.

3. Research gap identification

The various kinds of methods have been developed for the production of bio-diesel like, food catalytic cracking (FCC), hydro-treating-hydrocracking, utilization of biomass-derived synthesis gas (syn-gas) or hydrogen all these three methods are come under petroleum refinery (Huber & Croma, 2007). Another method was catalytic cracking of edible and non-edible vegetables the important issue in this method that the choice of catalyst and designing of reactors (Taufiqurrahmi & Bhatia, 2011). Other one is micro-emulsification, blending etc. but, finally we have selected the transesterification method. Bio-diesel is acquired by trans esterifying vegetable oils or different materials are to a great extent involved triglycerides with monohydric alcohols, most ordinarily methanol, to give the comparing mono-alkyl esters Figure-1. Subsequently, the unsaturated fat profile of bio-diesel relates to that of the parent oil or fat from which it is created. As the most economical liquor, methanol has been usually used to create bio-diesel. Accordingly, bio-diesel is frequently mentioned to as the unsaturated fat methyl esters (FAME) of a vegetable oil or creature fat or other triacylglycerol-containing material (Knothe & Razon, 2017). So, this method becomes most important for the production bio-diesel from edible and non-edible oils. Albeit both palatable and non-edible oils can be utilized for bio-diesel creation, eatable oils are broadly utilized right now. It has been assessed that 95% of the bio-diesel of the feed-stock comes from palatable oils. Changing over palatable oils into bio-diesel may negative influence the equilibrium of interest among food and fuel and lead to high food costs. to maintain a strategic distance from such issues, numerous scientists have examined a wide variety of plant oils to discover new and conceivable option for bio-diesel creation (Harne et al., 2012). So, we have chosen the *Calotropis procera* plants seeds for the production of bio-diesel the seeds of this plant have a great potential of oil. The *Calotropis procera is* a non-edible plant also. The oil extracted from *Calotropis procera* seeds with the help of Soxhlet apparatus. Along with it we had also find out that the Literature about this plant was very less as comparison other plants. So, we had selected this plant for the production of bio-diesel with the help transesterification. In the previous study we had seen the *Calotropis procera* plant used for the collection of its properties but in our present study

the actual conversion of bio-diesel from *Calotropis procera* seeds with the viewing of its properties.

At last, we had also found that the nanoparticls can also be synthesized from *Calotropis procera* milky latex. So, we decide to synthesize nanoparticls from its latex like iron, copper, and nickel all of these nanoparticls have their lot applications. These can also act as catalyst in the transesterification reaction for the creation of biomass. Anyway, a variety of different strategies has been created to get NPs of different shapes and sizes of iron, nickel, and Cu NPs including laser removal, gamma radiance, electron light, substance decrease, photochemical techniques, microwave handling, and warm deterioration of silver oxalate in water and in ethylene glycol, and organic manufactured strategy however the main parts tangled in the readiness of nanoparticles utilizing usual methodologies. Union of NPs utilizing plants separate is practicality, and consequently can be utilized as a financial and important option for the huge scope creation of NPs (Iravani, 2011). Literature survey revealed that *Calotropis procera* milky latex has been utilised by very few studies for the synthesis of nanoparticles.

4. Research objectives

The objective of my work is given as under: -

1. To synthesize the iron, copper and nickel nanoparticls from the milky latex of *Calotropis Procera* plant using green method.
2. To prepare bio-diesel from *Calotropis procera* plant seeds oil.
3. To describe the characteristics of iron, copper and nickel, nanoparticles by following techniques like UV-Visible spectroscopy, SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy), XRD (X-ray Diffraction), FTIR (Fourier Transform infrared spectroscopy)
4. Characterize the bio-diesel by using techniques like FTIR, (Fourier Transform Infra-red), GS-MS (Gas Chromatography-Mass Spectroscopy). In addition to this fuel properties of the bio-diesel will be examined by following parameters including cloud point, specific gravity, flash point, kinematic viscosity, refractive index, pour content, ash content, water content, calorific value and copper strip corrosion American Society for Testing and Materials (ASTM) procedures were adopted for analyzing the fuel properties.

5. Proposed methodology

In the literature review observed that many methods have been used for the synthesis of bio-diesel, like food catalytic cracking, micro emulsification, hydro-cracking, pyrolysis

etc. But we will use transesterification method for the production of bio-diesel and Soxhlet apparatus method for the extraction of oil from the seeds of *Calotropis procera*.

5.1. Materials

Seeds of *Calotropis procera*, methanol, sodium hydroxide, weighing machine, stirrer, beaker, conical flask, heating machine or burner, thermometer, filter paper, filtration apparatus, round bottom flask, tripod stand, oil of *Calotropis procera* seeds, Soxhlet apparatus, hexane, blender. The material for nanoparticls synthesised like, latex of *Calotropis procera* will be collected early in the morning. Whatman filter paper, copper nitrate, ferric sulphate, nickel acetate, distilled water, beaker, thermometer, conical flask, burner, methanol, burner, ethylene glycol, potassium hydroxide, magnetic stirrer etc.

5.2 Methods

5.2.1 Preparation of latex extract

Latex of *Calotropis procera* will be gathered promptly toward the beginning of the day. Rough latex will be acquired by cutting the green stems of *Calotropis procera* plants. Smooth white latex will be put away at 44°C temperature until use.

5.2.2 Synthesis of metal nanoparticles

In a standard reaction mixture, 1 mL rough latex will be diluted to 500 mL with refined deionized water to make it 0.5 %, and 20 ml of this latex solution will be combined with 20 mL solution of copper nitrate ,Ferric sulphate and Nickel acetate and the combination will allow you to stay at room temperature in a research facility while shaking nonstop. As copper nitrate, Ferric sulphate and Nickel acetate solutions interacts with the latex solution, within a short moment, uncontrolled decrease can occur, resulting in the union of monodisperse copper, iron and nickel nanoparticles (Harne et al., 2012).

5.2.3 Preparation of seeds/oil extraction

The seeds will be isolated from the floss physically then dried under the sun for 14h. A blender will be utilized to crush the seeds. The ground seeds will be broiler dried for 1h at 105°C temperature and afterward separated to get oil, utilizing hexane in a Soxhlet device at 60°C for 8h (Photo et al., 2014).

5.2.4 Methods for production of biodiesel

The transesterification reaction will be carried out in a 250 ml three-necked round bottom glass reactor with stirring, heating, and condensation capabilities. The reaction temperature (45 to 65°C), the molar ratio of oil and alcohol (1:3 to 1:12), the volume of iron nano catalyst (0.5 to 1.5 wt.%), the reaction time (60 to 180 min), and the stirring rate (100 to 500 rpm) will be optimized to achieve a high bio-diesel yield photosynthesized iron nano

catalyst (Bio- FeNPs), 1:9 molar ratio of oil and methanol, 65° C reaction temperature, and 150 min reaction time at 400 rpm were the optimal conditions. The above-mentioned optimized conditions for the development of bio-diesel will be used to conduct catalyzed transesterification of Calotropis procera seeds oil. Following the completion, the results of the reaction will be aloowed to cool to room temperature and stand for process segregation the excess methanol from the separated top will be removed. Rotary vacuum evaporator layer of crude bio-diesel (Rengasamy et al., 2016). The bio-diesel yield will be determined utilizing the accompanying condition: Bio-diesel yield% = amount of (FAME)(g)/ amount of oil used (g) × 100%.

6. Characterization of the extracted oil of *Calotropis procera* and nanoparticles

Nanoparticles from milky latex will be characterized by the following techniques like, scanning electron microscopy (SEM), UV-visible spectroscopy, (FTIR)Fourier-transform infrared spectroscopy (TEM) transmission electron microscopy, (XRD) X-ray diffraction, (FTIE) Fourier transform infrared spectroscopy and the bio-diesel will be characterized by (FTIR) Fourier transform infrared spectroscopy, (GS-MS) gas chromatography-mass spectroscopy.

7. Thesis outline

CHAPTER 1 (Introduction):

In the chapter we had introduced that how bio-diesel produced from *Calotropis procera* seeds oil through the transesterification method. The applications of nanoparticles also discussed here.

CHAPTER 2 (Literature Review):

In this chapter we had collected the lot of core information about bio-diesel by reading the old research paper related to bio-diesel and nanoparticles. And see that from which methods the bio-diesel produced as till.

CHAPTER 3 (Materials and Methods):

It includes the experimental details on material used, biological methods of synthesis, characterization techniques.

CHAPTER 4 (Results and Discussions):

In this chapter we had cleared our research objective that what we will do in our future research

Chapter 5 (Summary and Conclusions):

In the chapter 5 shows that which type of materials and methods used for the production of bio-diesel and nanoparticles.

Chapter 6 (References):

In this chapter references are elaborated.

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CERTIFICATE
(SYNOPSIS APPROVAL CERTIFICATE)

This is to certify that the Synopsis for Ph.D. Thesis titled, **"Preparation of biodiesel and synthesis of nanoparticles from *Calotropis procera* plant seeds"**, submitted by **Mr. Surinder Kumar** to the Career Point University, Hamirpur (H.P.), India in partial fulfillment of the requirements for the degree of **DOCTOR OF PHILOSOPHY** in the Department of Chemistry, **School of Basic and Applied Sciences** has been approved by the Research Advisory Committee (RAC) after an Oral Examination of the student on dated 04/03/2021.

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